



Acknowledgement:

Sample was kindly provided by Swedish National Forensic Centre (NFC), where the compound was identified. IN NFC structure has been verified by means of GC-MS, FT-IR and NMR. Original NFC`s report is available in EMCDDA EDND database. See at:

<http://www.emcdda.europa.eu/activities/action-on-new-drugs>

(password is required).

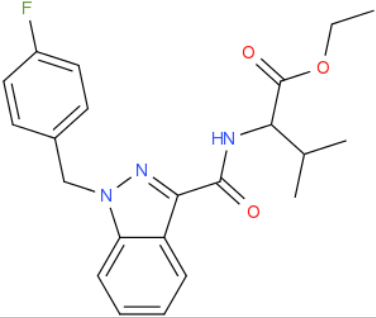
In the Slovenian National Forensic laboratory (NFL) sample was additionally analysed. The main goal of analyses performed in NFL-SI was to obtain FTIR-ATR and IR condensed spectra. NFL analytical report shown in this document.

ANALYTICAL REPORT¹ (NFL-SI)EMB-FUBINACA (C₂₂H₂₄FN₃O₃)

Ethyl 2-[[1-[(4-fluorophenyl)methyl]indazole-3-carbonyl]amino]-3-methyl-butanoate

Remark – other NPS detected: none

Sample ID:	1257-15
Sample description:	powder - white
Sample type:	collected /OTHER (kindly provided by Swedish NFC)
Date of sample receipt (M/D/Y):	8/24/2015
Date of entry (M/D/Y) into NFL database:	11/5/2015
Report updates (if any) will be published here:	http://www.policija.si/apps/nfl_response_web/seznam.php

Substance identified (in SFC)-structure ² (base form)	
Systematic name	Ethyl 2-[[1-[(4-fluorophenyl)methyl]indazole-3-carbonyl]amino]-3-methyl-butanoate
Other names	Ethyl (1-(4-fluorobenzyl)-1H-indazole-3-carbonyl)valinate
Formula (per base form)	C ₂₂ H ₂₄ FN ₃ O ₃
M _w (g/mol)	397,44
Salt form	base
StdInChIKey	YOECWCNJGHHTMN-UHFFFAOYSA-N
Compound Class	Cannabinoids
Other NPS detected	none
Add.info (purity..)	significant amounts of impurities were not detected by GC-MS, HPLC-TOF at NFL SI

¹ This report has been produced with the financial support of the Prevention of and fight against crime Programme of the European Union (grant agreement number JUST/2013/ISEC/DRUGS/AG/6413). The contents of this report are the sole responsibility of the National Forensic Laboratory and can in no way be taken to reflect the views of the European Commission.

² Created by OPSIN free tool: <http://opsin.ch.cam.ac.uk/> DOI: 10.1021/ci100384d

Report updates

date	comments (explanation)

Instrumental methods (if applied) in NFL

1. GC-MS (Agilent): GC-method is RT locked to tetracosane (RT=9.53 min). Injection volume 1 ml and split mode (1:50) . Injector temperature: 280 °C. Chromatographic separation: on column HP1-MS (100% dimethylpolysiloxane), length 30 m, internal diameter 0.25 mm, film thickness 0.25 mm. Carrier gas He: flow-rate 1.2 ml/min. GC oven program: 170 °C for 1 min, followed by heating up to 293 °C at a rate of 18 °C/min, hold for 6.1 min, then heating at 50 °C/min up to 325 °C and finally 2.8 min isothermal. MSD source EI = 70 eV. GC-MS transfer line T= 235°C, source and quadropole temperatures 280°C and 180°C, respectively. Scan range m/z scan range: from 50 (40) to 550 amu.

2. HPLC-TOF (Agilent): 6230B TOF with Agilent 1260 Infinity HPLC with binary pump, column: Zorbax Eclipse XDB-C18, 50 x 4.6 mm, 1.8 micron. Mobile phases (A) 0.1% formic acid and 1mM ammonium formate in water; (B) 0.1% formic acid in methanol (B). Gradient: starting at 5% B, changing to 40% B over 4 min, then to 70% over 2 min and in 5 min to 100%, hold 1 min and back to 5%, equilibration for 1.7 min. The flow rate: 1.0 ml/min; Injection volume 1 µl. MS parameters: 2GHz, Extended Dynamic range mode to a maximum of 1700 amu, acquisition rate 1.30 spectra/sec. Sample ionisation: by Agilent Jet Stream technology (Dual AJS ESI). Ion source: positive ion scan mode with mass scanning from 82 to 1000 amu. Other TOF parameters: drying gas (N2) and sheath temperature 325 °C; drying gas flow rate 6 l/min; sheath gas flow rate 8 l/min; nebulizer 25 psig; Vcap. 4000 V; nozzle 2000 V; skimmer 65 V; fragmentor 175 V and Octopole RF 750 V.

3.FTIR-ATR (Perkin Elmer): scan range 4000-400 cm⁻¹; resolution 4cm⁻¹

4. GC- (MS)-IR condensed phase (GC-MS (Agilent) & IR (Spectra analyses-Danny)

GC-method: Injection volume 1 ml and split mode (1:5). Injector temperature 280 °C. Chromatographic separation as above (1). Split MS : IR = 1:9.

MSD source EI = 70 eV. GC-MS transfer line T= 235°C, source and quadropole temperatures 280°C and 180°C, respectively. Scan range m/z scan range: from 50 (40) to 550 amu.

IR (condensed phase): IR scan range 4000 to 650, resolution 4 cm⁻¹.

5. IC (anions) (Thermo Scientific, Dionex ICS 2100), Column: IonPac AS19, 2 x 250mm; Eluent: 10mM from 0 to 10 min, 10-58 mM from 10 to 40min; Flow rate: 0.25 ml/min; Temperature: 30°C; Suppressor: AERS 500 2mm, suppressor current 13mA; Inj. Volume: 25 µl

Supporting information

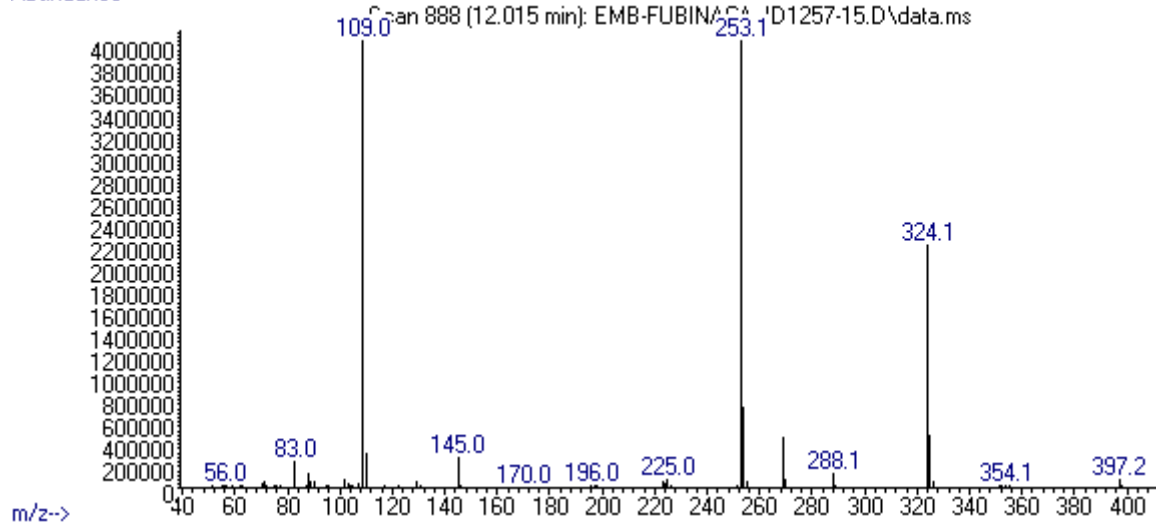
Solubility in	result/remark
CH ₂ Cl ₂	partially
MeOH	soluble
H ₂ O	

Analytical technique:	applied	remarks
GC-MS (EI ionization)	+	NFL GC-RT (min): 12,02 BP(1): 253; BP(2): 109,BP(3) :324,
HPLC-TOF	+	Exact mass (theoretical): 397,1802; measured value Δppm:0; formula:C22H24FN3O3
FTIR-ATR	+	direct measurement
FTIR (condensed phase) always as base form	+	extract in CH ₂ Cl ₂
IC (anions)	+	
NMR	-	<i>structure has been verified by Swedish National Forensic Centre</i>
validation		
other		

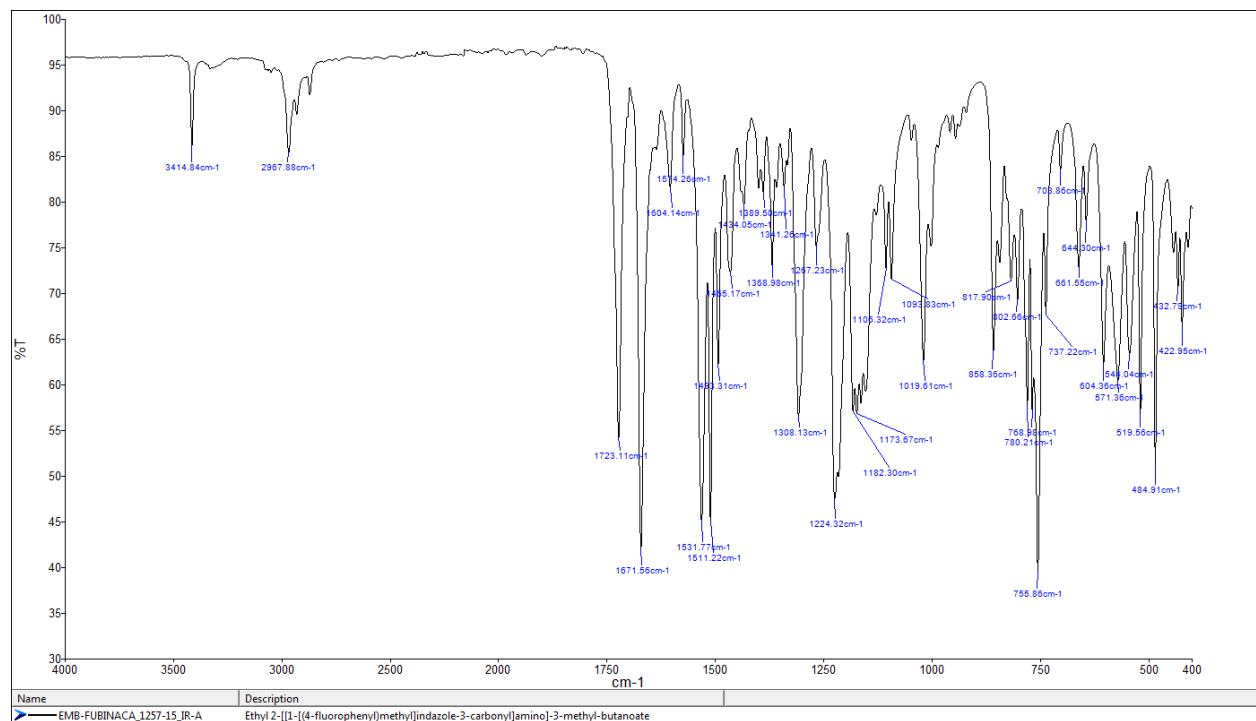
ANALYTICAL RESULTS

MS (EI)

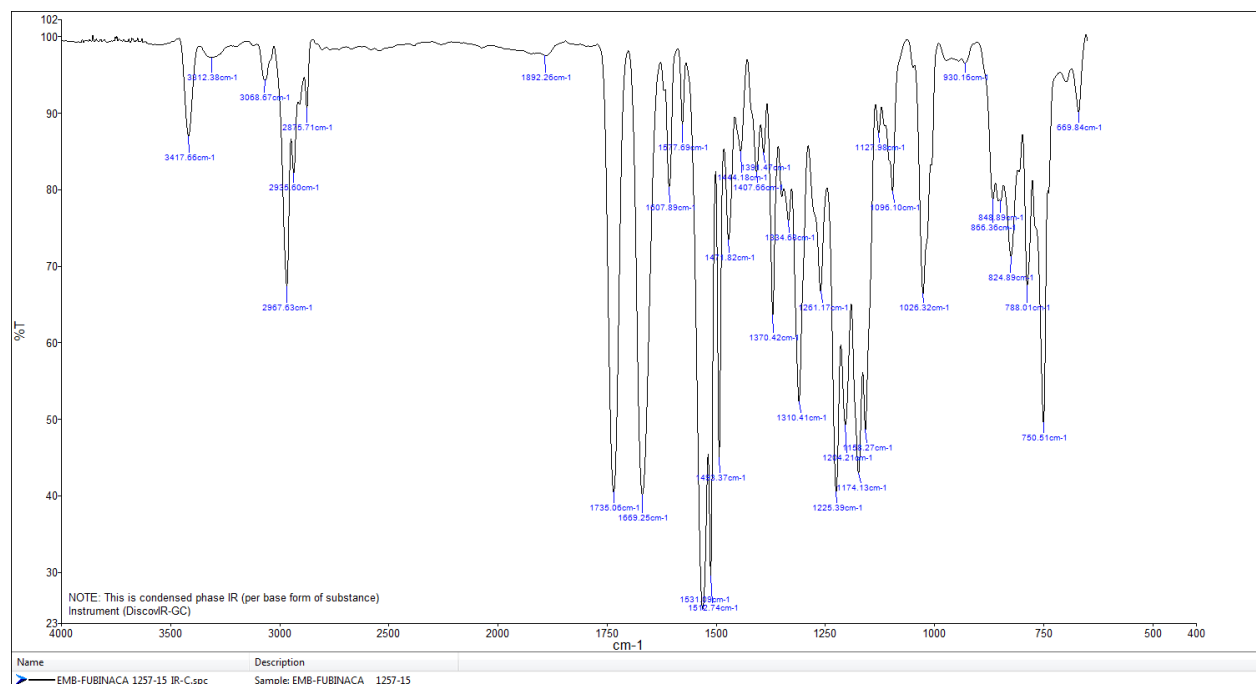
Abundance



FTIR-ATR - direct measurement



IR (condensed phase)



Target Compound Screening Report

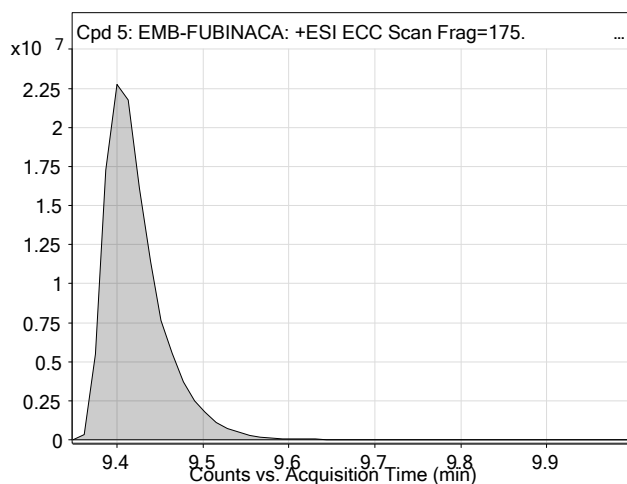
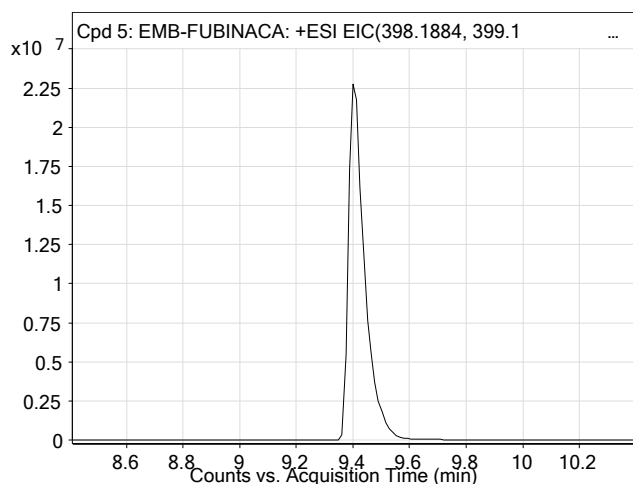
Data File	EMB-FUBINACA_1257-15_TOF.d	Sample Name	EMB-PINACA
Sample Type	Sample	Position	P1-C3
Instrument Name	6230B TOF LC-MS	User Name	TG
Acq Method	droge general-13-5-2015-XDB-C18-ESI-poz.m	Acquired Time	9/2/2015 5:17:31 PM
IRM Calibration Status	Success	DA Method	Droge_Default.m
Comment	extract in MeOH		

Compound Table

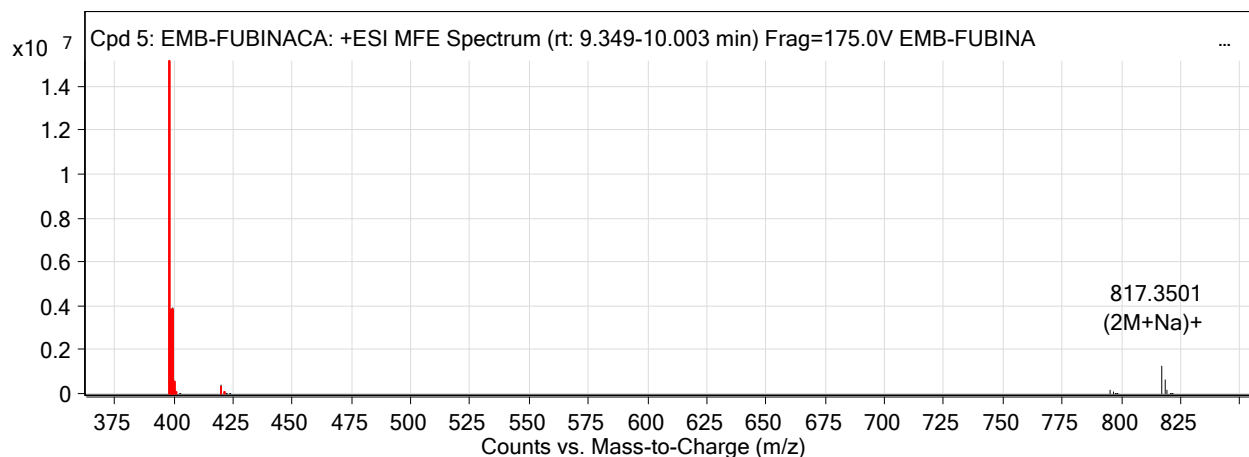
Label	Tgt Name	MFG Formula	Obs. RT	Obs. Mass
Cpd 5: EMB-FUBINACA	EMB-FUBINACA	C22 H24 F N3 O3	9.408	397.1802

Name	Obs. m/z	Obs. RT	Obs. Mass	DB RT	DB Formula	DB Mass	DB Mass Error (ppm)	Find Cpds Algorithm
EMB-FUBINACA	398.1874	9.408	397.1802	9.408	C22 H24 F N3 O3	397.1802	0	Find by Molecular Feature

Compound Chromatograms



MFE MS Zoomed Spectrum



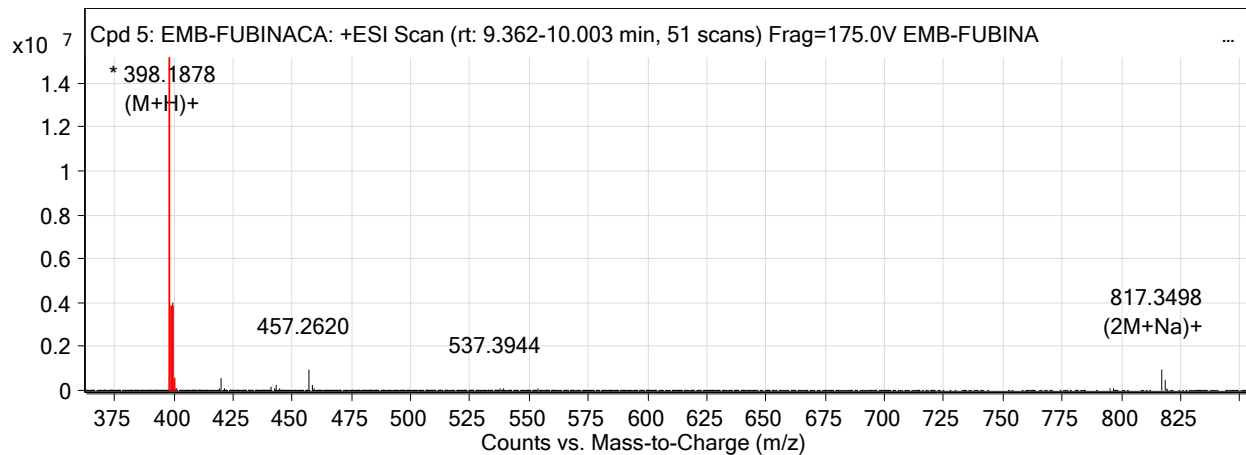
MS Spectrum Peak List

Obs. m/z	Charge	Abund	Formula	Ion/Isotope
398.1874	1	15165631	C22 H24 F N3 O3	(M+H)+
399.1906	1	3890611.09	C22 H24 F N3 O3	(M+H)+
400.194	1	518099.51	C22 H24 F N3 O3	(M+H)+
420.1697	1	359106.31	C22 H24 F N3 O3	(M+Na)+
421.1724	1	83558.13	C22 H24 F N3 O3	(M+Na)+
795.3677	1	156055.97		(2M+H)+
796.3704	1	75584.9		(2M+H)+

Target Compound Screening Report

817.3501	1	1247199.5	(2M+Na)+
818.3535	1	609024.58	(2M+Na)+
819.3559	1	153369.35	(2M+Na)+

MS Zoomed Spectrum



--- End Of Report ---

Peak Integration Report

Sample Name:	EMB-FUBINACA_1257-15_IC	Inj. Vol.:	25,00
Injection Type:	Unknown	Dilution Factor:	1,0000
Program:	ANIONI	Operator:	kemija
Inj. Date / Time:	27-okt-2015 / 22:11	Run Time:	42,00

No.	Time min	Peak Name	Peak Type	Area $\mu\text{S}^*\text{min}$	Height μS	Amount n.a.
TOTAL:				0,00	0,00	0,00

